# COMPONENTS:

- (1) Terbium chloride; TbCl<sub>3</sub>; [10042-88-3]
- (2) Ethanol; C<sub>2</sub>H<sub>6</sub>O; [64-17-5]
- (3) Water; H<sub>2</sub>0; [7732-18-5]

#### ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. <u>1976</u>, 21, 551-4; Russ. J. Inorg. Chem. (<del>Engl</del>. Transl.) <u>1976</u>, 21, 296-8.

# VARIABLES:

Temperature

# PREPARED BY:

T. Mioduski and M. Salomon

#### EXPERIMENTAL VALUES:

solubility of TbCl<sub>3</sub>.6H<sub>2</sub>O in 96.8 % C<sub>2</sub>H<sub>5</sub>OH<sup>a</sup>

	sample 1	sample 2	sample 3	sample 4	mean solub	ilities
t/°C	g/100 g <sup>b</sup>	g/100 g	g/100 g	g/100 g	g/100 g	mol kg <sup>-10</sup>
20	30.75	30.68	30.50	30.61	30.63	1.183
30	29.94	30.03	30.32	30.06	30.08	1.152
40	30.31	30.49	30.33	29.94	30.27	1.163
50	30.84	30.81	30.77	30.92	30.83	1.194
60	32.92	33.03	32.65	32.63	32.80	1.307

 $<sup>^{\</sup>mathrm{a}}$ It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS: TbCl3.6H2O prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over CaCl<sub>2</sub>, P<sub>2</sub>O<sub>5</sub> and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 158.0 - 158.8°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr CuSO4 followed by distn. Ethanol concn detd refractometrically and pycnometrically.

#### ESTIMATED ERROR:

Soly: results apparently precise to within  $\pm$  0.9 % (compilers).

Temp: nothing specified.

bSolubilities reported as grams of hexahydrate in 100 g of solvent.

<sup>&</sup>lt;sup>C</sup>Molalities calculated by the compilers.

					273
COMPONENTS:			ORIGINAL	MEASUREMENTS	:
(1) Terbium chloride [10042-88-3]	; TbCl <sub>3</sub> ;		Kirmse,		
(2) Alkoxy-ethanols			Tr. II V 1971, 20	ses. Konf. po 0–6.	o Teor. Rastvorov
VARIABLES:			PREPARED	BY:	
T/K = 298			T. Miodu	ski and M. Sa	alomon
EXPERIMENTAL VALUES:					
			TbCl <sub>3</sub> so	lubility <sup>a</sup>	
solvent			mass %	mol kg <sup>-1</sup>	nature of the solid phase
2-methoxyethanol;	с <sub>3</sub> н <sub>8</sub> о;	[109-86-4]	3.8	0.15	$TbC1_3.nC_3H_80_2$ (n = 2-3)
2-ethoxyethanol;	c4H10O2;	[110-80-5]	11.9	0.509	TbC13.2C4H1002
		AUXILIARY	INFORMATI	ON	
METHOD/APPARATUS/PROC Experimental details probably similar to pauthor which are comp volume.	not given, b	ks of the	Nothing work by probably and Cart	the authors prepared by er (1).	MATERIALS: ut based on previous the anhydrous salt was the method of Taylor
			ESTIMATE		
			Nothing	specified.	
			REFERENCE 1. Taylo J. In	r, M.D.; Car	ter, C.P. hem. <u>1962</u> , 24, 387.

# COMPONENTS: (1) Terbium chloride; TbCl<sub>3</sub>; [10042-88-3] (2) 1,2-Diethoxyethane; C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>; [629-14-1] VARIABLES: T/K = 298 ORIGINAL MEASUREMENTS: Kirmse, E.M.; Zwietasch, K.J. Z. Chem. 1967, 7, 281.

#### EXPERIMENTAL VALUES:

The solubility of TbCl3 in 1,2-diethoxyethane at 25°C was reported to be

0.22 mass %

The corresponding molality calculated by the compiler is

0.0083 mol kg<sup>-1</sup>

The composition of the solid phase was given in terms of the Tb:Cl:ether ratio as

1:2.93:1.37

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over  $P_2O_5$ .

Tb was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

# SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of  $C_2H_5I$  with the monoethylether of ethylene glycol.

#### ESTIMATED ERROR:

No estimates possible.

#### REFERENCES:

Taylor, M.D.; Carter, C.P.
 J. Inong. Nucl. Chem. <u>1962</u>, 24, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Terbium chloride; TbCl <sub>3</sub> ; [10042-88-3]	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J Oelsner, L.; Niedergesaess, U. Z. Chem. 1968, 8, 472-3.
(2) Ethers	Kirmse, E.M. Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.
VARIABLES:	PREPARED BY:
Room temperature: T/K around 298	T. Mioduski and M. Salomon

#### EXPERIMENTAL VALUES:

			$TbCl_3$ solubility $^{\mathtt{a,b}}$		
solvent			mass %	mol kg <sup>-1</sup>	
1-ethoxy-2-methoxyethane;	c5H12O2;	[5137-45-1]	0.6	0.023	
1,4-dioxane;	с <sub>4</sub> н <sub>8</sub> 0 <sub>2</sub> ;	[123-91-1]	0.3	0.011	

<sup>&</sup>lt;sup>a</sup>Molalities calculated by the compilers.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isother-mally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Tb was determined by complexometric titration.

No other details given.

# SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

#### ESTIMATED ERROR:

Nothing specified.

# REFERENCES:

Taylor, M.D.; Carter, C.P.
 J. Inorg. Nucl. Chem. <u>1962</u>, 24, 387.

bNature of solid phases not specified.

1	ORIGINAL MEASUREMENTS:		
bium chloride; TbCl <sub>3</sub> ; 042-88-3]	Kirmse, E.M.; Dressler, H.		
yl ethers	Z. Chem. <u>1975</u> , 15, 239-40.		
: perature (293-298 K)	PREPARED BY: T. Mioduski and M. Salomon		
	bium chloride; TbCl <sub>3</sub> ; 042-88-3] yl ethers		

# EXPERIMENTAL VALUES:

			$solubility^a$		
solvent			mass %	mol kg <sup>-1</sup>	
1-methoxybutane;	°5 <sup>H</sup> 12 <sup>O</sup> ;	[628-28-4]	0.23	0.0087	
1-methoxypentane;	°6 <sup>H</sup> 14 <sup>O</sup> ;	[628-80-8]	0.7	0.027	
1-methoxyheptane;	с <sub>8</sub> н <sub>18</sub> 0;	[629-32-3]	3.3	0.129	
1-methoxyoctane;	с <sub>9</sub> н <sub>20</sub> 0;	[929-56-6]	1.5	0.057	
1-methoxynonane;	c <sub>10</sub> H <sub>22</sub> 0;	[7289-51-2]	2.75	0.107	
1-methoxydecane;	с <sub>11</sub> н <sub>24</sub> 0;	[7289-52-3]	1.9	0.073	

 $<sup>^{</sup>m a}$ Molalities calculated by the compilers. Compositions of solid phases were not specified.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing  $P_4O_{10}$ . To was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS: Nothing specified.

# ESTIMATED ERROR:

Nothing specified.

#### ORIGINAL MEASUREMENTS: COMPONENTS: Terbium chloride; TbCl3; Korovin. S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N. [10042-88-3] Zh. Neorg. Khim. 1975, 20, 908-14; Russ. Tributylphosphate; C12H27O4P; (2) J. Inorg. Chem. (Engl. Transl.) 1975, 20. [126-73-8] 508-11. VARIABLES: PREPARED BY: T/K = 298T. Mioduski and M. Salomon

#### EXPERIMENTAL VALUES:

# Composition of saturated solutions

mass %	mol/kg sln	g dm <sup>-3</sup>	mol dm <sup>-3</sup>	mol kg-l (compiler)	density/g cm <sup>-3</sup>
38.5	1.45	525.0	1.98	2.36	1.36

The solid phase is  $TbCl_3$ 

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH3. The pptd Tb(OH)3 was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous, and only the anhydrous TbCl3 was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and TbCl<sub>3</sub> in solution.

# SOURCE AND PURITY OF MATERIALS:

Anhydrous TbCl<sub>3</sub> prepared by chlorination of the oxide with CCl<sub>4</sub> vapor (1,2). Source and purity of materials not given. Tb was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

# ESTIMATED ERROR:

No estimates possible.

- Korshunov, B.G.; Drobot, D.V.;
   Bukhtiyarov, V.V.; Shevtsova, Z.N.
   Zh. Neorg. Khim. 1964, 9, 1427.
- Novikov, G.I.; Tolmacheva, V.D. Zh. Prikl. Khim. 1965, 38, 1160.

# Terbium Chloride 278 COMPONENTS: ORIGINAL MEASUREMENTS: (1) Terbium chloride; TbCl<sub>3</sub>; [10042-88-3] Kirmse, E. M. Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6. (2) Alkyl amines VARIABLES: PREPARED BY: T/K = 298T. Mioduski and M. Salomon EXPERIMENTAL VALUES: TbCl<sub>3</sub> solubility<sup>a</sup> mass % mol kg-1 solvent 0.644 sec-C<sub>4</sub>H<sub>11</sub>N; [13952-84-6] 14.6 2-butanamine; (sec-C<sub>4</sub>H<sub>q</sub>)<sub>2</sub>NH; [626-23-3] 0.15 0.0057 di-2-butylamine; <sup>a</sup>Molalities calculated by the compilers. AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE: Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume. Nature of solid phases not specified. SOURCE AND PURITY OF MATERIALS: Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1). ESTIMATED ERROR: Nothing specified. REFERENCES: 1. Taylor, M.D.; Carter, C.P.

J. Inorg. Nucl. Chem. 1962, 24, 387.

#### COMPONENTS: ORIGINAL MEASUREMENTS: Terbium chloride; TbCl<sub>3</sub>; Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, [10042-88-3] N.A.; Zhilina, T.A. Zh. Neorg. Khim. <u>1977</u>, 22, 1761-6; Russ. J. Inorg. Chem. (Engl. Transl.) <u>1977</u>, 22, (3) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$ ; [680-31-9] 955-8. VARIABLES: PREPARED BY: Room temperature: $T/K = 298 \pm 3$ T. Mioduski and M. Salomon

#### EXPERIMENTAL VALUES:

Starting with the solvate  $TbCl_3.3((CH_3)_2N)_3PO$ , the solubility at 25  $\pm$  3°C<sup>a</sup> was given as

 $0.128 \text{ mol dm}^{-3}$ 

 $^{
m a}$ Table 3 in the English translation of the source paper states the temperature to be  $23 \pm 3^{\circ}$ C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 (  $t_1$  = 169 d). Authors state that results for  $^2$ both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be тьс1<sub>3</sub>.3С<sub>6</sub>H<sub>18</sub>N<sub>3</sub>ОР.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

# SOURCE AND PURITY OF MATERIALS:

TbCl3.3C6H18N3OP prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P205 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR: Soly: precision  $\pm$  0.007 mol dm $^{-3}$  at a 95% level of confidence (authors).

Temp: precision ± 3K.

# REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. Elektrokhimiya 1975, 11, 163.

COMPO	NENTS:	ORIGINAL MEASUREMENTS:	
(1)	Terbium chloride; TbCl <sub>3</sub> ; [10042-88-3]	Lyubimov, E.I.; Batyaev, I.M.	
(2)	Tetrachlorostannate; SnC1 <sub>4</sub> ; [7646-78-8]	Zh. Prikl. Khim. <u>1972</u> , 45, 1176-8.	
(3)	Phosphorus oxychloride; POCl <sub>3</sub> ; [10025-87-3]		
	BLES: = 293	PREPARED BY:	

EYPE	RIMENT	AT. VAT	HES:

Concentration of SnC14

SnCl <sub>4</sub> :POCl <sub>3</sub> ratio	SnCl <sub>4</sub> concentration	solubility/mo	les Tb dm <sup>-3 a,b</sup>	
(by volume)	mol dm <sup>-3</sup>	Tb203	ть <sub>4</sub> 0 <sub>7</sub>	
1:250	0.035	0.069		
1:100	0.085	0.30	0.056	
1:50	0.17	0.15 (0.13)	0.18 (0.015)	
1:25	0.33	0.067	0.059	
1:15	0.59	0.051	0.040	
1:10	0.78	0.018	0.047	

T. Mioduski

$$Tb_2O_3 + 6POCl_3 = 2TbCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of  ${ t TbCl}_3$  is enhanced by complex formation according to

 $2TbCl_3 + 3SnCl_4 = Tb_2(SnCl_6)_3$ 

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method used. POCl<sub>3</sub> + SnCl<sub>4</sub> solutions were prepared by volume in a dry box. The SnCl<sub>4</sub> content was verified by chemical analysis for Sn. This solution and Tb<sub>2</sub>O<sub>3</sub> were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20°C to 2 hours.

Tb was determined by colorimetric analysis or the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

#### SOURCE AND PURITY OF MATERIALS:

Tb407 of "the first sort" was reduced to Tb203 with hydrogen at 950°C.

"Pure" grade  $SnC1_4$  and  $POC1_3$  were dehydrated with  $P_20_5$  and distilled under vacuum.

# ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably  $\pm$  0.2 K (compiler).

<sup>&</sup>lt;sup>a</sup>Solutions preheated to 220°C. Values in parenthesis correspond to preheating at 120°C.

<sup>&</sup>lt;sup>b</sup>This is also the solubility of TbCl<sub>3</sub> in the SnCl<sub>4</sub>-POCl<sub>3</sub> mixture because the oxide is quantitatively converted to the chloride according to